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A LOW-DENSITY POTTING COMPOUND

by

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ABSTRACT

A combined total of 4 years development effort and production experience has conclusively proven the value of a glass-microballoon-filled epoxy resin system in potting applications where weight saving, without a drastic sacrifice in physical properties, or resistance to high-level mechanical shock is a prime requirement.

A LOW-DENSITY POTTING COMPOUND

Introduction

The development of certain electronic packages for missile and airborne applications has created a need for a lightweight potting compound that offers greater environmental protection than that provided by the available polyurethane foams. Preliminary efforts indicated that this objective could be best achieved by incorporating low-density fillers into conventional epoxy resins.

Several such fillers[1] were investigated during the course of this effort, but none possessed processing characteristics and end properties equal to those of glass microballoons. An exhaustive review of possible resin systems was required as this type filler proved more difficult to handle than conventional solid fillers. It is not interchangeable with other fillers and cannot be freely substituted into existing formulations since it must be processed over rather limited ranges of time, temperature, and viscosity. Failure to maintain these conditions yields nonhomogeneous castings or mixes which are too viscous to process.

Development

It should be borne in mind that the formulation developed is intended for use in the encapsulation of complex, expensive, electronic circuitry designed to

[1] Urea-formaldehyde, phenolic, and silica microballoons as well as polystyrene and aluminum silicate hollow spheres of a much larger particle size were investigated.

withstand extremely severe environments. Therefore, a difficult-to-process material is not objectionable when it yields an end product of the high confidence level required.

The system finally selected parallels a previous Sandia system using the standard filler mica in that diethanolamine-cured Epon 828 was utilized for many of the same reasons that made it attractive earlier. Several of these reasons are:

1. good pot life
2. fluidity at the processing temperature
3. low exotherm
4. essentially nontoxic

A processing study of the microballoon-filled mix showed that other resins of a similar epoxide equivalent are not all interchangeable since differences in viscosity and reactivity result in less homogeneous castings.

Other variables, in addition to the choice of resin and hardener, were necessarily fixed to ensure homogeneous mixes. The concentration of filler was fixed at its maximum processable limit; anything less results in thinner mixes which permit floatation of the hollow spheres. The cure temperature was firmly fixed at 150°F; lower temperatures enhanced floatation because the mix was in a fluid state for a longer time. Increased temperatures also enhanced floatation since the viscosity of the mix was sharply reduced. Batch size was also restricted as small batches lose heat readily and become too viscous to process. Large batches exhibit unusually high exotherms because of the thermal insulation imparted by the hollow spheres; this results in a reduced pot life. Timing is very important if the mix is to be successfully evacuated during periods of optimum viscosity. The type of mixer and time of mixing must also be defined to avoid excessive crushing of the fragile microballoons.

These investigations of the properties of the several raw materials and their interaction within a mix resulted in the recommendation of a single

formulation and a tightly defined process.[2] This single formula is the one which yielded the maximum advantages of the glass microballoon filler consistent with a minimum of processing limitations.

From the foregoing, it can be seen that the processing of a microballoon-filled epoxy resin is critical. If such a critical process is to remain manageable, reasonable control must be exercised over the individual constituents of the formulation. Fortunately, the resin and hardener have given little difficulty. However, the glass microballoons have sometimes exhibited serious batch-to-batch variations that have led to processing difficulties or a reduction in end properties of the cured mix.

When these batch variations became evident, a program was set up to make several properties measurements on those batches on hand and to continue the measurements on all future batches.[3]

That property of greatest importance is average particle density. Since the formulation is based on parts by weight, any change in particle density is reflected by an adverse change in viscosity due to the variation in the volume of filler incorporated. Particle size distribution is also important since it controls the packing of individual particles within the resin matrix. Moisture content is the last requirement considered mandatory and is controlled because excessive moisture results in undesired agglomeration of the filler particles. Additional properties presently being determined on an "Information Only" basis are alkalinity, water solubility, and floatation ratio.

Cold-shock tests are being run by Sandia on all lots of microballoons received. This test is not part of the microballoon specification, since it must be evaluated from the final mix rather than from the microballoons alone. The

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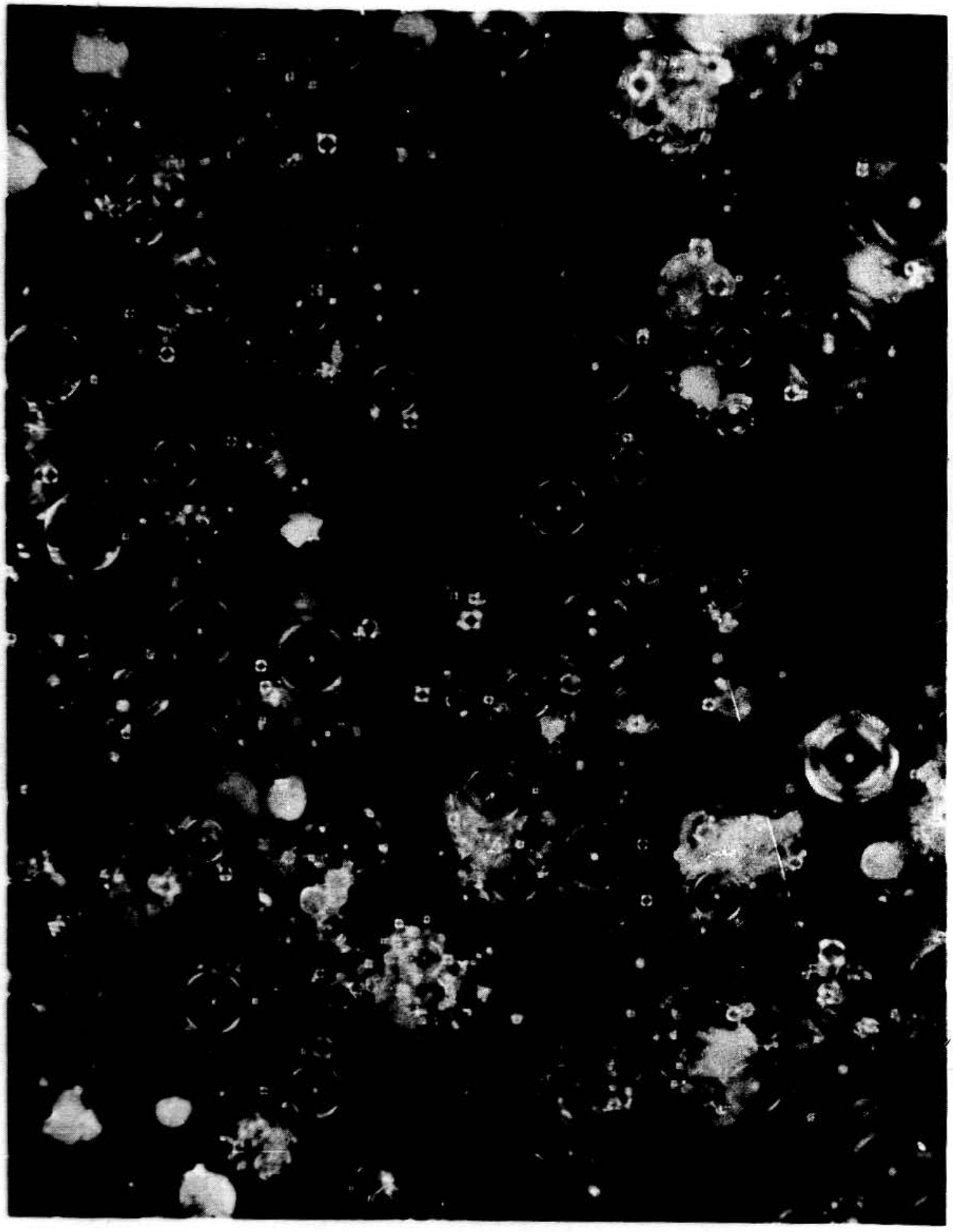
[2] See Addendum No. 1.

[3] Standard Oil of Ohio developed and originally produced this material. It is now being produced under exclusive license from Sohio by Emerson and Cuming, Inc., as Eccospheres-R.



Eccospheres-R 100X

tests are primarily intended to ensure that a processable mix which will pass thermal and mechanical shock testing can be guaranteed. A continuing investigation is under way to determine what properties and what limits of these microballoon properties best define this objective.

Eccospheres-R Glass Microballoons Properties Chart

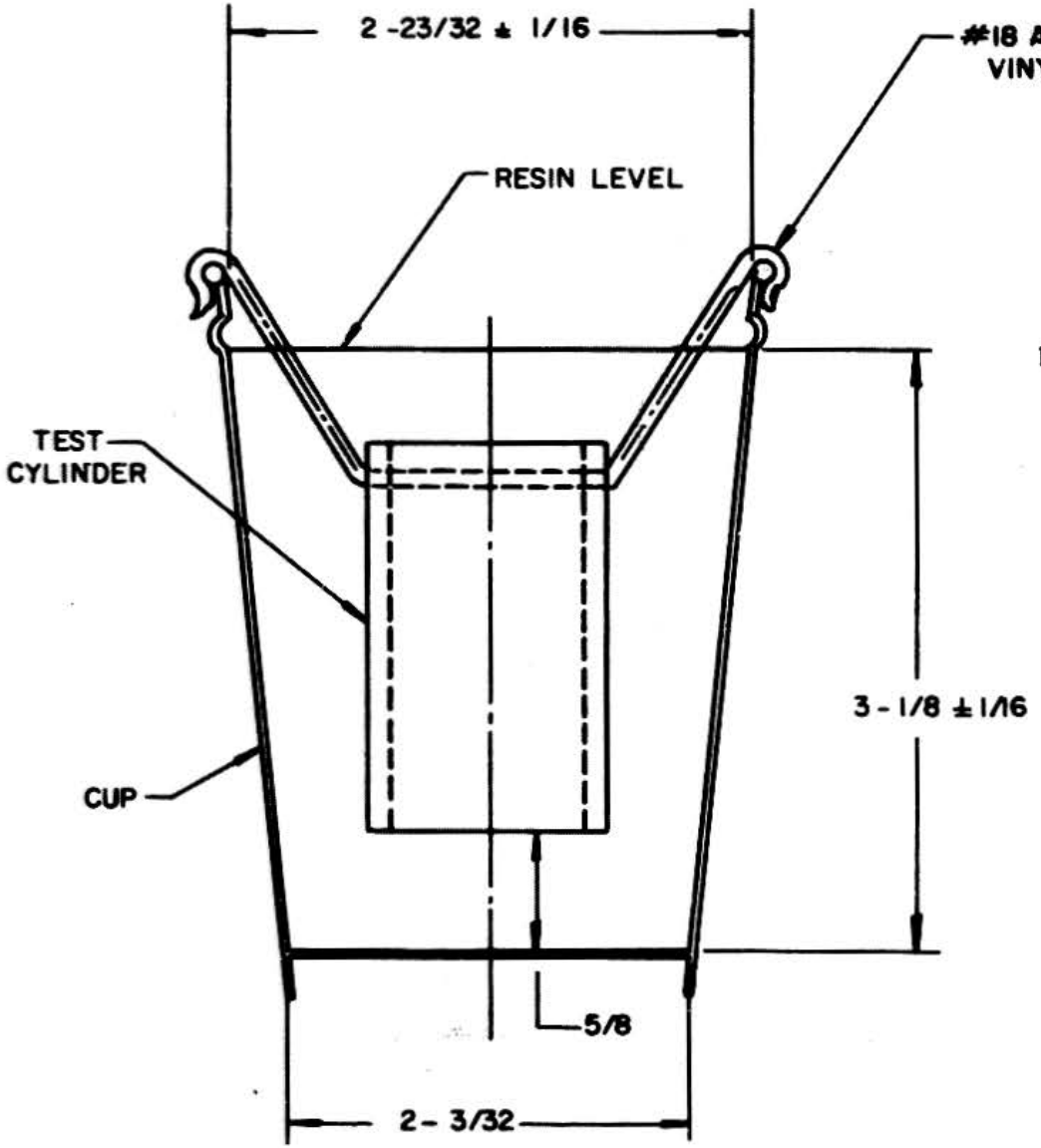
Date	9/59	10/59	11/59	11/59	12/59	1/60		3/60	8/60	1/61	
Batch	20-S	20-S	20-S	21-S	21-S	23-S		24-S	27-S	30-S	Spec.
Sieve analysis, wt. %	RT	RT	RT	RT	RT	RT	CM	CM	CM	CM	
+60 mesh (250 microns)	0.1	0.0	0.0	0.1	0.0	0.0	0.1	0.2	0.1	0.3	0.5 max.
-200 mesh (47 microns)	47.8	24.3	61.2	37.2	25.4	25.0	29.9	34.1	41.1	38.4	20-50
Avg. particle density, gm/cc	0.48	0.48	0.44	0.49	0.35	0.45		0.46	0.45	0.45	0.39-0.46
Moisture content, wt. %	<0.05	<0.05	<0.05	<0.05	0.09	0.02		0.04	0.10	0.06	0.20 max.
Alkalinity, meq/gm	5.3	5.0	5.1	5.0	5.0	4.8		5.1	4.9	4.8	
Water solubility, wt. %	41	32	42	35	37	32		41	40	37	
Floatation ratio*	9-1	7-1	11-1	6-1	9-1	6-1		9-1	9-1	9-1	
Thermal shock***	2P	2P	2P	0P	**	5P		5P	4P	5P	
	1F	1F	1F	3F		0F		0F	0F	0F	

P = Passed
F = Failed
RT = Ro-Tap shaker
CM = Cenco-Meinzer shaker

*Volume ratio of floaters to sinkers in water
**Too viscous to pour
***Surface of test specimen improved and number of test specimens increased for greater reliability starting January 1960.

The study of the properties of early batches of glass microballoons led to the issuance of a tentative specification.[4] From the data listed on the microballoon properties chart, it can be seen that these more recent batches generally meet the limits established in the specification and yield processable mixes that pass thermal shock testing.

[4]See Addendum No. 2.

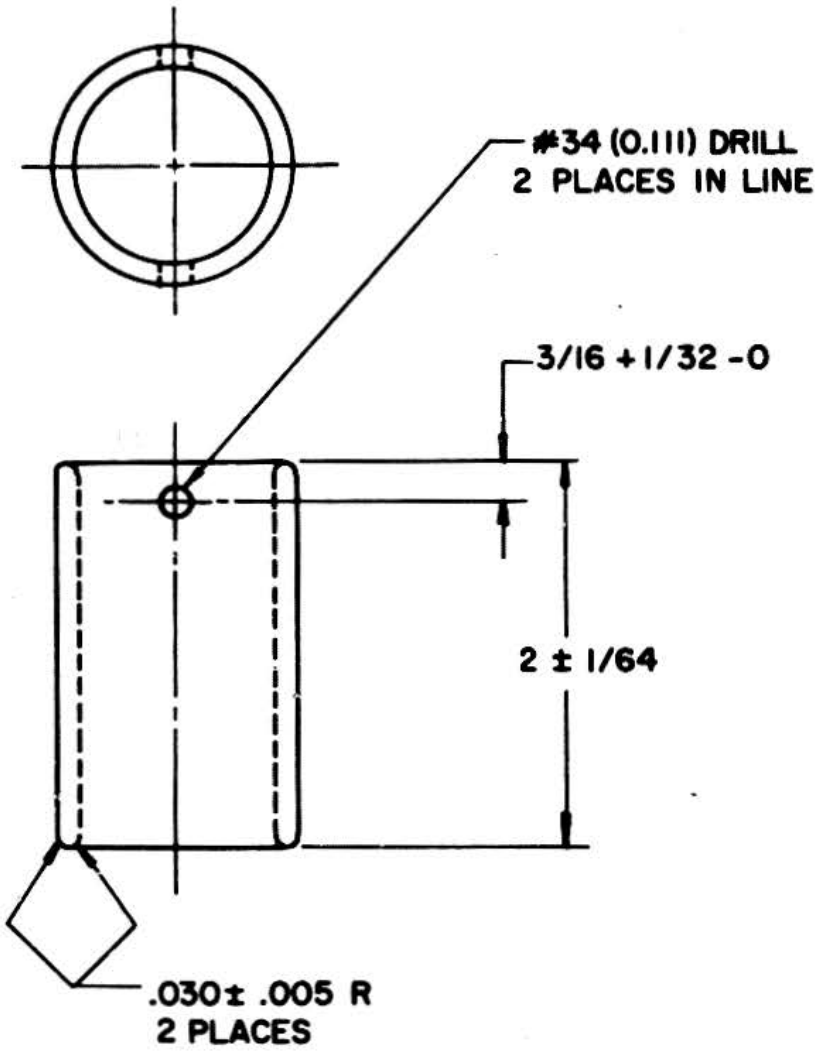


Notes:

1. **Maintain concentricity of cup and cylinder within 1/32 in.**
2. **Maintain other tolerances to ± 1/32 in. except as shown.**
3. **A container which fits these requirements is the 8 ounce Dixie No. 2108 or the 8 ounce Dixie "Mira-Glaze", #4338. The container shall be removed following cure.**
4. **Subject the embedded specimen three times to the following cycle: 4 hours at room temperature (75° ± 10°F) immediately followed by 4 hours at -65°F, then immediately return to room temperature. Visually inspect for cracks following each cycle.**

(Dimensions in inches)

Cold Shock Test, Assembly, and Test Conditions



(Dimensions in inches)

Notes:

1. **Scale: Full size**
2. **Material: Seamless steel tubing 1-1/4" OD x .120 wall, low carbon, MIL-S-11486 cold drawn.**
3. **Finish: Machine or polish to obtain a bright surface of 50 microinch rms inside and outside.**

Cold Shock Test, Test Cylinder

Low-Density Potting Compound Properties

		-65°F	Room temp.	+165°F
Tensile				
ASTM D638	Maximum stress, psi	5,480	4,660	2,200
	Strain at max. stress, %	1.1	1.1	2.6
	Modulus, x 10^5 psi	5.3	4.4	1.8
Compressive				
ASTM D695	Maximum stress, psi	14,600	10,600	3,480
	Strain at max. stress, %	3.9	3.3	10.6
	Modulus, x 10^5 psi	4.4	4.2	0.9
Flexural				
ASTM D790	Maximum stress, psi	7,230	5,850	2,920
	Strain at max. stress, %	1.6	1.4	4.6
	Modulus, x 10^5 psi	4.7	4.4	2.0

Thermal coefficient of expansion ASTM D696	Temp. range (°C)	Expansion rate x 10^{-6} in/in/°C
	-60 to 0	35
	0 to 40	39
	40 to 70	42

Thermal conductivity BTU-in/hr/ft^2/°F	1.1
Specific gravity ASTM D792	0.85
Heat distortion temp. 264 psi ASTM D648	170°F
Heat resistance, wt. loss, % MIL-I-16923	0.04
Moisture absorption, wt. % MIL-I-16923	0.37
Fungus resistance	Nonnutrient
Dielectric constant ASTM D150	10^3 cps 3.02 10^6 cps 2.72
Dissipation factor, % ASTM D150	10^3 cps 3.96 10^6 cps 2.55

Low-Density Potting Compound Properties (continued)

Dielectric strength, vpm ASTM D149	430	
Volume resistivity, ohm-cm ASTM D257	Room temp.	2.05×10^{14}
	150°F	1.97×10^{13}
	200°F	9.30×10^{10}
	250°F	2.43×10^{9}
	300°F	3.68×10^{8}

Comparative Properties of Various Density Potting Compounds

	Polyurethane foam*	Glass-microballoon-filled epoxy	Mica-filled epoxy**
Density, lbs/ft^3	10	53	102
Tensile strength, psi	350	4,660	7,700
Tensile modulus, x 10^5 psi	0.1	4.4	12
Compressive strength, psi	325	10,650	17,970
Compressive modulus, x 10^5 psi	0.1	4.2	10
Flexural strength, psi	450	5,850	11,300
Flexural modulus, x 10^5 psi	0.2	4.4	12

*Average properties for several types of 10 lb/ft^3 foam, that density most commonly used by Sandia.

**50 pbw Epon 828
50 pbw 1000 mesh mica
6 pbw diethanolamine

An observer, upon studying the specification and the properties chart, might question the broad limits on the sieve analysis. However, all attempts to narrow this range and to include information on further sieve sizes have resulted in a mass of confused data, almost all of which apply equally well to acceptable microballoons.

The data for average particle density have proven to be more valuable. It can be seen that Batch 21-S, dated December 1959, exhibited a particle density of 0.35 gm/cc. Because of this low density, the formulation which is based on parts by weight was too viscous to pour, since it contained an abnormally large volume of filler. Conversely, Batch 21-S, dated November 1959, exhibited an average particle density of 0.49 gm/cc which is above that allowed by the specification. This material was rejected; it yielded castings that completely failed the thermal shock test since the mix did not contain a sufficient volume of filler. This discrepancy within one batch has led to a better system of defining and identifying batches.

Batches 27-S and 30-S have both exhibited peculiar characteristics that emphasize the need for further studies attempting to define acceptable microballoons. The average particle density is determined by measuring the density of the cured casting. Knowing the density of the cured resin, it is a simple calculation to determine the average particle density of the included balloons. These measurements are made on hand-stirred mixes to eliminate any variable due to crushing of the balloons by mechanical mixing. Batches 27-S and 30-S, when incorporated into the usual 70 resin - 30 microballoon weight ratio, proved to be too viscous to pour and therefore indicated a too-low average particle density. However, when measured, they were both found to be 0.45 gm/cc near the high end of the acceptable range. Nevertheless, these microballoons when mixed mechanically (yielding an average particle density of 0.51 gm/cc) proved to be quite processable and gave acceptable end properties. Earlier batches had been carefully mixed mechanically and showed an average increase of only 0.02 gm/cc. This larger increase for Batches 27-S and 30-S

may indicate that these are more fragile balloons. Why they exhibit a high particle density and yet yield viscous mixes has yet to be explained.

To date nothing significant has been noted in the data accumulated for those properties measured for "Information Only." The alkalinity of each batch has been observed because it is well known that the reaction of the epoxy cure is base catalyzed. The values for water solubility appear alarmingly high, but the electrical properties of the cured mix are relatively unaffected after exposure to high humidity due to the protection offered by the resin matrix. The ratio of particles that float in water to those that sink does not seem to affect processability or thermal shock resistance within the limits observed.

In addition to Eccospheres-R[5], Emerson and Cuming also produces refined grades of microballoons with improved chemical, temperature, and moisture resistance. Unfortunately, the refining process reduces the range of the particle distribution and eliminates all high-density "sinkers." The greater range of particle distribution for Eccospheres-R, although admittedly quite variable, results in castings that are superior in cold shock resistance to those containing refined microballoons. Furthermore, the elimination of "sinkers" in castings processed and cured per Addendum No. 1 results in an area at the bottom that is devoid of filler. Therefore, it is desirable to have a certain concentration of high-density particles to offset the floatation of the low-density particles. As a result of these shortcomings, it is felt that Eccospheres-R are better suited to the needs of this program.

Of the other low-density fillers examined, all proved inferior to glass microballoons for one or more reasons. Generally, these can be described as undesirable particle size or distribution, lack of uniformity, fragility, high viscosity mixes, or poor cold shock resistance. Phenolic microballoons are being re-examined and, because of the processing experience that has been gained, they now appear much more promising. The phenolic balloons have a considerable price advantage, but when one considers the confidence and

[5]The "R" indicates raw product.

reliability that have been established for the glass microballoon system in such critical applications, then raw materials costs become relatively insignificant.

Characteristics

Capabilities

1. The use of this microballoon-filled epoxy resin can result in a considerable weight saving where the weight of the encapsulating material is an appreciable part of the total weight of a package. The specific gravity of the cured system averages 0.85 as opposed to 1.64 for a general-purpose system.

2. There is presently no dynamic test available to permit a quantitative evaluation of the high-level shock resistance of this material. However, components encapsulated with it have survived an estimated 5000 g, with a 2-millisecond rise time, whereas the use of conventional materials has led to complete failures under these conditions.

3. In addition to its excellent mechanical shock resistance, this material also possesses superior thermal shock resistance. To date, it is the only rigid system to pass Sandia's most severe thermal shock test.

Limitations

1. To achieve maximum properties, the system has been loaded to the point where its viscosity imposes a processing problem. Therefore, the electronic package designer must take this factor into account and should assign work only to experienced potting organizations.

2. Because of the thermal insulating nature of the microballoon filler, the cure exotherm frequently reaches a peak of 190°F. Furthermore, processing characteristics presently preclude the possibility of a reasonably short-time, low-temperature cure for thermally sensitive components, as is possible with general-purpose systems.

Applications

With careful design consideration, this resin system can be incorporated into electronic packages of the type that are currently being encapsulated with the more familiar filled resins. However, the advantages in a particular application must outweigh the disadvantages listed above.

ADDENDUM NO. 1

PROCESS FOR ENCAPSULATION OF
ELECTRICAL COMPONENTS

1. SCOPE

1.1 This process designates the requirements for encapsulating electrical components in a low-density filled epoxy resin.

2. REQUIREMENTS

2.1 Formulation -- The encapsulating compound shall be formulated from the materials listed below taken in their exact weights. The tolerance on weights of materials shall be ±1 percent. The approved materials satisfactory for use in this process are listed in Section 3.1.

Epoxy resin	700 grams
Filler	300 grams
Hardener	85 grams

2.1.1 Master Batches -- Master batches of filled epoxy resin may be prepared by combining 70 parts, by weight, of epoxy resin with 30 parts of filler. The final formulation shall then be prepared by combining 1000 grams of this mix with 85 grams of hardener.

2.1.2 Filler Material -- Since the filler material tends to segregate and stratify in its shipping container during shipping and handling, it shall be blended, but not sieved or screened, before use to ensure a uniform particle distribution throughout the material. The filler shall be a free-flowing powder at time of use.

2.1.2.1 When any filler is removed from a new drum of material, a sufficient quantity of bagged activated desiccant shall be inserted in the inner polyethylene bag to maintain dryness, and the polyethylene bag shall be tightly resealed each time it is opened.

2.2 Mold and Component Preparation -- The mold (or assembly housing if the mold remains as a part of the finished unit) and electrical components shall be preheated in a forced convection oven to a temperature of 175±5°F at the time

of pouring the encapsulating compound. When not an integral part of the assembly, the mold shall be coated with a thin even film of a suitable mold-release agent and allowed to dry (see 3. 1. 5).

2. 3 Mixing of Encapsulating Compound -- For best results during the subsequent deaeration step, it is recommended that the container have a diameter approximately equal to its height and a volume at least four times the volume of the mixed compound. Combine the epoxy resin and filler and mix thoroughly in a planetary-action mixer with a flat beater until the filler has been wetted and thoroughly dispersed throughout the resin. The mixing operation shall be completed in approximately 10 minutes to avoid excessive crushing of the filler. (A Hobart mixer has been found satisfactory.)

2. 3. 1 Heat the resin-filler mixture of 2. 3 above to 150±5°F in a forced convection oven and add the hardener. Mix thoroughly in equipment described in 2. 3 above until the mixture is uniform throughout (approximately 3 minutes).

2. 4 Deaeration of Encapsulating Compound -- Immediately upon completion of mixing, the mixture shall be returned to the 150°F oven. Eight minutes ±30 seconds after addition of hardener, the mix shall be evacuated at a pressure of 1-3 mm Hg absolute. Continue the evacuation for 1 minute after the initial foam rise collapses. The entire evacuation process shall take a maximum of 3 minutes. The deaerated resin mix shall be used in the subsequent steps as soon as practicable.

2. 5 Pouring of Encapsulating Compound -- At the time of pouring, the encapsulating compound shall be at a temperature of 180 to 190°F, preferably not lower than 185°F. The preheated components and mold shall be partially filled with the warm, deaerated encapsulating compound. Degas the mold and components by placing them in a vacuum chamber and evacuating at a pressure of 1-3 mm Hg absolute and maintaining this reduced pressure for at least 3 minutes. Allow pressure to return to atmospheric. In no instance shall this step be completed later than 30 minutes after the addition of hardener (2. 3. 1).

2. 5. 1 In some units the configuration and spacing of components may be such that in order to remove all entrapped air it will be necessary to introduce the encapsulating compound in two steps, evacuating as above after each step.

2. 5. 2 Add additional encapsulating compound to bring level up to height indicated on the product drawing or to 1/4 to 1/8 inch of level, if unit is to be topped as specified in 2. 5. 3. For this last step, the encapsulating compound may be utilized for as long as 40 minutes after addition of hardener. No further evacuation is necessary. Any bubbles which appear at the surface of the filled mold prior to gelation of the encapsulating compound shall be broken by spraying the surface with resin defoamer (3. 1. 4).

2.5.3 Topping -- If shrinkage of the potting compound is such as to leave an uneven surface, it may be desirable to apply a topping. When a topping is to be applied, the mold or housing shall be filled to within 1/4 to 1/8 inch from the top. The compound shall be permitted to gel by heating in a forced convection oven for a minimum of 3 hours and not more than 6 hours at a temperature of 150±5°F. After the compound has gelled, the mold shall be filled to the required level with encapsulating compound. For the topping operation the encapsulating compound may be utilized for as long as 40 minutes after addition of hardener (Section 2.3.1).

2.6 Cure -- The filled housing or mold shall be placed in a forced convection oven maintained at a temperature of 150±5°F and shall be cured at that temperature for a minimum of 24 hours.

3. NOTES

3.1 Sources of Supply

3.1.1 Epoxy Resin -- The only epoxy resin approved for use in this process is:

Epon 828	Shell Chemical Corporation 500 Fifth Avenue New York 18, New York

3.1.2 Filler Material -- The filler used in this process consists of expanded glass beads. The only approved source is:

Eccospheres-R Glass Microballoons	Emerson and Cuming, Inc. 869 Washington Street Canton, Massachusetts

3.1.3 Hardener -- The only hardener approved for use in this process is diethanolamine. It is available from the following sources:

Primary sources

Diethanolamine	Union Carbide Chemicals Co. 2770 Leonis Boulevard Los Angeles, California
Diethanolamine	Dow Chemical Company Midland, Michigan

Secondary sources

Diethanolamine Cat. No. 1598	Distillation Products Industries Division Eastman Kodak Company Rochester 3, New York

Diethanolamine Cat. No. D-45	Fisher Scientific Company 2850 S. Jefferson Street St. Louis 18, Missouri

3.1.4 Resin Defoamer -- A satisfactory resin defoamer is:

Resin defoamer No. 1	Par Industries, Inc. 2193 E. 14th Street Los Angeles 21, California

3.1.5 Mold Release -- A mold release agent which has been found satisfactory when casting in a separate mold is:

Garan 225	Ram Chemical Company Gardena, Califorr a

3.2 The ingredients of this encapsulating compound may be toxic. Hence, adequate ventilation should be provided in the handling of these materials to prevent undue exposure. Ingestion or skin contact with these materials shall be avoided. If accidental skin contact should occur, the exposed areas should be washed immediately with soap and water.

ADDENDUM NO. 2

SPECIFICATION FOR
GLASS MICROBALLOON FILLER MATERIAL

1. SCOPE

1.1 Scope -- This specification designates the requirements for glass microballoons used as a filler for encapsulating resins.

2. REQUIREMENTS

2.1 Form -- The filler shall be a white, free-flowing powder and shall be free from any visible contaminants or foreign materials.

2.2 Sieve Analysis -- When tested, as specified in Section 3.3.1, the sieve analysis of the filler material shall be as follows:

a. No more than 0.5 percent by weight of filler shall be retained on a 60 mesh sieve.

b. No less than 20 percent nor more than 50 percent by weight of filler shall pass through a 200 mesh sieve.

2.3 Water Content -- The water content of the microballoon filler shall not be greater than 0.20 percent by weight when determined as specified in Section 3.3.2.

2.4 Particle Density -- The average particle density of the filler material shall by within the range of 0.39 to 0.46 gram per cubic centimeter when determined as specified in Section 3.3.3.

3. QUALITY ASSURANCE PROVISIONS

3.1 Lot -- A lot of microballoon filler material shall be: (1) that quantity made during one production run from one batch of ingredients and offered for delivery at one time or (2) a homogeneous blend of two or more lots described in (1) above. Each lot shall be identified to distinguish it from all other lots.

3.2 Sampling -- At least one entire shipping container of microballoon filler material shall be randomly selected from each lot.

3.2.1 Blending -- All the filler material from the sample container shall be placed in a tumbler and tumbled until the particles are uniformly blended (maximum of 20 minutes). After blending, a representative sample of filler material shall be taken for testing, and the remaining filler material shall be immediately returned to its original package. A sufficient quantity of activated bagged desiccant with a suitable humidity indicator shall be placed with the filler material in the polyethylene bag, and the container shall be tightly closed. The test sample shall be stored in a dry, airtight container.

3.3 Test Procedures

3.3.1 Sieve Analysis

3.3.1.1 Apparatus

a. Balance accurate to 0.1 gram.

b. Cenco-Meinzer sieve shaker.

c. 60 mesh and 200 mesh sieves, cover and pan conforming to ASTM E-11.

3.3.1.2 Procedure

a. Weigh out a 100±0.1 gram sample of filler.

b. Assemble the clean sieves with the 60 mesh on top, followed by the 200 mesh, and with the pan at the bottom of the series. Transfer the sample to the 60 mesh sieve.

c. Place the covered sieve series on the shaker and shake for 20 minutes at shaker setting of "6."

d. Separate the nested sieves and pan. Weigh each sieve and pan and its contained filler material to the nearest 0.1 gram. Carefully remove the residue and clean each sieve. Reweigh the cleaned sieves and pan. The difference in weights will equal the percentage of residue on each sieve or pan.

NOTE: Sample recovery from the sieves and pan shall total at least 98 grams.

3.3.2 Water Content

3.3.2.1 Apparatus

a. Drying oven - capable of maintaining a temperature of 250±5°F.

b. Analytical balance.

c. Platinum dish - 100 ml capacity.

d. Desiccator.

3.3.2.2 Procedure

a. Heat a clean 100-ml platinum dish in a drying oven for 2 hours at 250±5°F. Remove the dish and cool in a desiccator. Tare the dish.

b. To the platinum dish add an approximate 10-gram microballoon sample, accurately weighed, to the nearest milligram.

c. Place sample and dish in the drying oven for 2 hours at 250±5°F. Remove from oven, cool to room temperature in a desiccator, and then weigh.

3.3.2.3 Calculation

$$\text{Percent water content} = \frac{W - B \times 100}{W}$$

where W = original weight of sample

B = sample weight after drying.

3.3.3 Particle Density

3.3.3.1 Apparatus

a. 25 x 150 mm test tubes

b. Fisher chain gravitometer

3.3.3.2 Sample Preparation

3.3.3.2.1 Formulation of Casting Compound -- Tolerance on weights of materials shall be ±1 percent.

Epoxy resin	70 grams
Microballoon filler	30 grams
Diethanolamine	8.5 grams

3.3.3.2.2 Mold Preparation -- Clean the test tubes and coat the inside surface with a thin film of mold release. Allow to dry.

3.3.3.2.3 Mixing and Deaeration of Casting Compound

a. Combine the epoxy resin and filler and mix thoroughly by hand until the filler has been wetted and uniformly dispersed throughout the resin.

b. Heat the resin-filler mixture to 150±5°F in a forced convection oven and then add the hardener. Mix thoroughly until the resultant mix is uniform (approximately 3 minutes).

c. Immediately upon completion of mixing, return the mixture to the 150°F oven. After 10 minutes have elapsed from the addition of hardener (step b, above), remove the mix from the oven, place it in a suitable vacuum chamber, and evacuate to a pressure of 1 to 3 mm Hg absolute. Continue the evacuation for 1 minute after the initial foam rise collapses. The entire evacuation process shall be completed within 3 minutes.

3.3.3.2.4 Pouring of Casting Compound

a. Immediately after evacuation pour the mix into the prepared test tube mold, filling to a level approximately 1-1/2 inches from the top.

b. Degas the mold and casting compound by placing it in a vacuum chamber and evacuating to a pressure of 1 to 3 mm Hg absolute and maintaining this reduced pressure for at least 3 minutes.

c. Add additional casting compound to bring level up to the top of the test tube. No further evacuation is necessary.

3.3.3.2.5 Cure -- Place the filled mold in a forced convection oven maintained at a temperature of 150±5°F and cure at that temperature for at least 24 hours.

3.3.3.3 Test Specimens

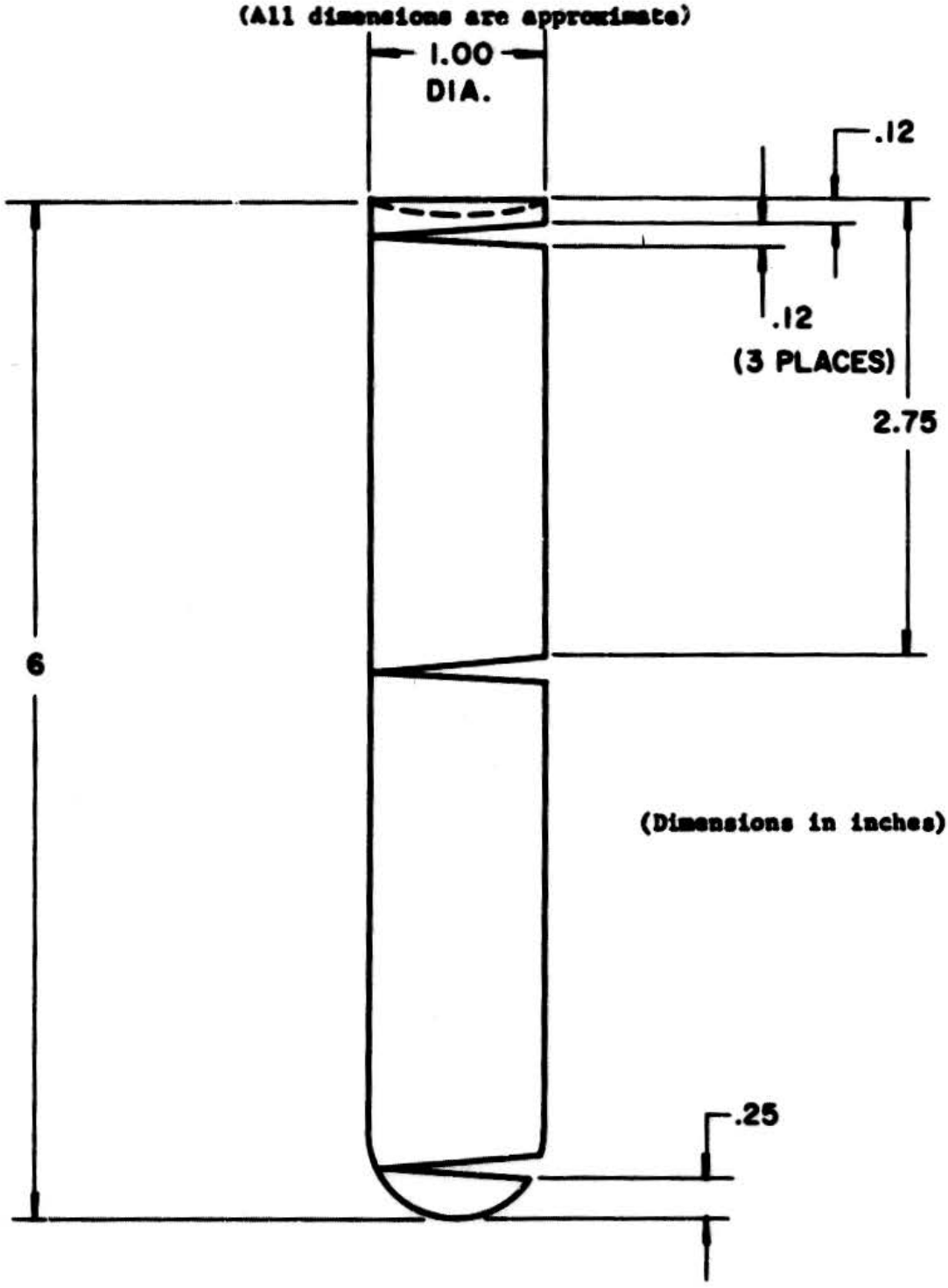
a. Remove the casting from the mold.

b. Take three wedge-shaped specific gravity test specimens from the casting as shown in the following illustration. Specimens shall be free of entrapped bubbles.

c. Using a fine abrasive, sand or grind the flat surfaces smooth. Remove dust by brushing with a stiff-bristled fiber brush.

d. Determine density of each specimen by means of the gravitometer. The specimen shall be held by wedging it in the underside of one of the slots of the gravitometer basket. A small quantity of wetting agent shall be added to the water to facilitate release of bubbles.

3.3.3.4 Particle Density Calculation -- The average particle density shall be calculated as follows:



Specific Gravity Specimen Locations

$$\text{Average particle density} = \frac{30}{\frac{108.5}{D} - 65.42}$$

where D = average density of three specimens.

3.3.3.5 Note -- If the nature of the filler material is such that a 70-30 resin-filler mix is not pourable, then a 75-25 resin-filler mix shall be prepared, and the following changes shall be made in the determination.

a. Section 3.3.3.2.1

Epoxy resin	75 grams
Microballoon filler	25 grams
Diethanolamine	9 grams

b. Section 3.3.3.4 $\text{Average particle density} = \frac{25}{\frac{109}{D} - 70}$

3.4 Acceptance Tests -- Each lot of microballoon filler shall be tested for average particle density and water content and shall be subjected to a sieve analysis. If any specimen fails to meet requirements of this specification, then the lot represented by that specimen shall be subject to rejection.

3.5 Certification -- Each lot of filler material shall be certified as to water content, sieve analysis, and average particle density.

4. PREPARATION FOR DELIVERY

4.1 Packaging -- The microballoon filler material shall be packaged in 5-pound and in 25-pound-size fiber containers having polyethylene-bag liners.

4.1.1 Marking -- The container shall be labeled with the manufacturer's name, product designation, lot number, and date of manufacture of individual lots, as defined in 3.1.

5. NOTES

5.1 Sources of Supply -- The only microballoon filler material qualified under this specification is:

Eccospheres-R glass microballoons	Emerson and Cuming, Inc. 869 Washington Street Canton, Massachusetts

EVALUATION OF MICROBALLOON FILLER MATERIAL "FOR INFORMATION ONLY"

1. GENERAL

1.1 Intended Use -- This document is intended for use in conjunction with "Specification for Glass Microballoon Filler Material," for the gathering of additional engineering information. It is not used for procurement.

2. REQUIREMENTS

2.1 Properties to be Evaluated -- Each lot sample obtained in accordance with Section 3.2 of the above specification shall be analyzed or tested for the following properties:

Leachable alkalinity
Water solubility
Floatation ratio

At least three specimens shall be taken and analyzed for the determination of each property.

3. TEST METHODS

3.1 Alkalinity

3.1.1 Apparatus

a. Electric heater.

b. Erlenmeyer flask - 250 ml with 24/40 ground-glass top.

c. Reflux condenser, water cooled - with 24/40 ground-glass joint.

d. Burette - 50 ml graduated.

3.1.2 Reagents

a. Hydrochloric acid, standardized, 0.1N solution.

b. Phenolphthalein indicator - 0.1 percent solution in methyl alcohol.

3.1.3 Procedure

a. Place an approximate 1-gram sample of microballoon filler, accurately weighed to the nearest milligram, in a 250-ml Erlenmeyer flask. Add 100 ml distilled water and connect the flask to a water-cooled reflux condenser.

b. Heat the water to boiling and allow to reflux for 2 hours. Allow flask to cool until it can be handled. Disconnect flask from reflux condenser.

c. Add several drops of phenolphthalein indicator solution and titrate to the pink end point with standard 0.1N HCl. Record the titer obtained.

d. Connect the flask to the condenser again and continue to reflux for an additional hour. Remove flask from the condenser and titrate to the end point again. Record the titer.

e. Continue refluxing the sample for 1-hour periods, titrating after each period until the titer for an individual titration is less than 1 ml of 0.1N HCl. (This point is usually reached at the end of the fourth or fifth reflux period.) Record the total quantity of HCl used for all the titrations.

IMPORTANT! Save the flask and sample for the determination of solubility of microballoons in water.

3.1.4 Calculation

$$\text{Alkalinity in meq. HCl/gram} = \frac{A \times N}{W}$$

where A = total ml HCl used in titrations

N = normality of HCl

W = weight of sample in grams.

3.2 Solubility

3.2.1 Apparatus

a. Filter flask - 500 ml, connected to water aspirator.

b. Filtering crucible - low form, fritted glass, medium porosity, 30 ml.

c. Walter crucible holder.

d. Analytical balance.

e. Drying oven - capable of maintaining a temperature of 250±5°F.

3.2.2 Reagents -- Methyl alcohol, reagent grade.

3.2.3 Sample -- The sample for this determination is contained in the flask retained from the determination of alkalinity.

3.2.4 Procedure

a. Place a tared, filtering crucible in a crucible holder of the filtering flask and start the water aspirator.

b. Transfer the contents of the flask to the filtering crucible. Wash the flask several times with methyl alcohol. (The fine particles of filler are more easily transferred by alcohol than by water.)

c. Wash the filter well with water, followed by several washes with alcohol.

d. Place the crucible and insoluble residue in an oven and dry at 250±5°F for 2 hours. Remove the crucible and allow to cool to room temperature in a desiccator. Weigh the crucible when cool.

3.2.5 Calculation

$$\text{Solubility, percent} = \frac{(W - A) \times 100}{W}$$

where W = original weight of microballoon sample

A = weight of insolubles filtered.

3.3 Floatation Ratio

3.3.1 Apparatus

a. 250-cc graduated cylinder.

3.3.2 Procedure

a. Place 100 cc of microballoon filler in the 250-cc graduate.

b. Fill the graduate to 250 cc with water and shake well to wet the microballoons.